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**FACTORS INFLUENCING CORROSION PROTECTION
PROVIDED BY SOLID FILM LUBRICANT COATINGS**



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TECHNICAL REPORT

By

G. P. Murphy

F. S. Meade

December 1965

**U. S. ARMY WEAPONS COMMAND
ROCK ISLAND ARSENAL
RESEARCH & ENGINEERING DIVISION**

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Laboratory Branch

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ABSTRACT

This investigation included the effect of (1) resin-pigment ratio, (2) coating thickness, and (3) wearing away of the film on the corrosion protection provided by solid lubricant coatings. These factors were investigated as possible approaches to improvement of the corrosion inhibition provided by MIL-L-46010(MR) type solid film lubricants. This improved corrosion inhibition was to be obtained at no sacrifice in wear life.

Two series of solid film lubricants of various resin-pigment ratios were made using good and poor pigment dispersion techniques. These series of solid film lubricants were evaluated at several levels of coating thickness. Corrosion protection was evaluated by means of 20% Salt Fog cabinet while wear life was determined by the Falex Wear Test method. A combination friction-salt fog test was used in determining the effect of wearing away of the film on corrosion resistance.

It was found that a solid film lubricant can be manufactured, using good pigment dispersion techniques, which will provide 1000 hours of salt fog protection. This lubricant showed no loss in wear life. It was also found that, by using a 0.0010 in. coating made up of two layers, salt fog life can be increased by at least 6 fold without sacrificing wear life.

The investigation also pointed up the fact that wearing away of the solid lubricant coating decreases the effectiveness of the coating as a corrosion inhibiting barrier.

FOREWORD

This investigation was carried out under DA Project No. 1C024401A107 and AMS Code 5025.11.801. The project title is "Lubricants, Friction and Wear." The title of the particular phase under which the study was carried out is "Corrosion Protection Provided By Solid Film Lubricants."

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PROBLEM

To study factors that may influence the corrosion protection provided by solid film lubricant coatings.

To increase the corrosion protection provided by Specification MIL-L-46010(MR) solid film lubricants without sacrificing wear life.

BACKGROUND

In 1957 work was begun at the Rock Island Arsenal Laboratory on the evaluation of the then available commercial resin bonded type of solid film lubricants. This work reported by Meade and Murphy⁽¹⁾ in 1958 showed that a solid film lubricant consisting of an epoxy resin pigmented with molybdenum disulphide had the best overall wear, friction and extreme pressure characteristics. At the time of the report, corrosion was not regarded as a problem, therefore, the corrosion protecting characteristics of the solid film lubricants were not evaluated.

Following this investigation work began on the development of a solid film lubricant with long wear life. This work culminated in 1960 with the development by Murphy and Meade⁽²⁾ of a solid film lubricant superior to the available commercial materials. This lubricant, RIA Compound 9A, was formulated without consideration of corrosion preventing properties as the need for such a solid film lubricant was not recognized at that time.

However, corrosion protection suddenly became a problem of considerable importance when the results of a salt fog test on a missile launcher which had been extensively lubricated with RIA Compound 9A was made known. Johnson et al⁽³⁾ reported that all parts coated with solid film lubricant rusted badly during the 168 hour salt fog test.

A corrosion protecting solid film lubricant was, therefore, developed by Meade and Murphy⁽⁴⁾ in 1961 that was a modification of RIA Compound 9A. The modification consisted of removing graphite, which had been shown to be a corrosion accelerator, from the formulation and adding an acid acceptor, dibasic lead phosphite, to combat the acid formed by the hydrolysis of the molybdenum disulphide.

This latest formulation covered by Army Specification MIL-L-46010(MR) gave 120 hours of salt fog protection and 500 minute Falex Wear life when applied to zinc

phosphatized steel. These results are much better than those obtained for materials meeting the Navy-Air Force Specification MIL-L-8937(ASG) which covers the same class of solid film lubricants.

However, it was thought this protection was still inadequate as the best protection against corrosion was obtained by using a cadmium plated steel substrate. It was shown by Calhoun et al⁽⁵⁾ that the use of the cadmium plate substrate drastically reduced the wear life of solid film lubricants under high bearing load conditions.

It was thought, therefore, if the corrosion preventing properties of a solid film lubricant could be sharply increased without reducing its wear life the need for the cadmium plate substrate would be eliminated. This would result in longer wear life of the lubricated parts and would decrease the cost of solid film lubricant application.

APPROACH

It would seem that the best approach to long corrosion protection would be to increase the efficiency of the solid lubricant coating as a physical barrier to penetration by moisture, acids, salt fog and other corrosive atmospheres. This could possibly be accomplished by either (1) increasing the resin content of the film or (2) increasing the thickness of the coating or both. The amount of resin added or the amount of film thickness increase will be limited by the effect on the wear life of the coating. Crump⁽⁶⁾ and others have shown that wear life of solid film lubricants is dependent on both film thickness and resin to pigment ratio. Wear life increases with increasing film thickness and increased amount of resin in the coating up to a point, after which a drastic reduction occurs.

The effect of an increase in resin content and also an increase in film thickness was evaluated according to the following procedure. All test specimens were grit blasted and zinc phosphatized prior to application of the coating. The thickness of the coating for the resin content study was 0.0005 in. and for the film thickness study several film thicknesses were used. The coatings for the resin content study were cured for one hour at 400°F. For the film thickness study, the coating was only partially cured between application of the different coats, this of course was done prior to the final cure of one hour at 400°F.

Falex Wear life was determined for the formulations made up with varying resin content and also for the various

film thicknesses by the standard wear life test method. This method is Method 3807 of Federal Test Method Standard 791a. Corrosion tests were also run on the various formulations and film thicknesses. The corrosion tests were run in accordance with Method 6061 of Federal Test Method Standard 141. In this manner the amount of corrosion protection provided at the maximum level of wear life could be determined.

In addition to the studies of the effect of resin-pigment ratio and film thickness on corrosion protection provided by solid film lubricants, the effect of wear on corrosion protection was also investigated. Panels coated with solid film lubricant were subjected to reciprocating sliding against one another under light load and low speed on the Cincinnati Stick Slip Tester. The coefficient of friction was determined and the panels were subjected to 72 hours in the 20% Salt Fog cabinet. At the end of the 72 hour exposure, the panels were inspected for evidence of corrosion and the coefficient of friction was again determined. This procedure was continued until a 100% increase in coefficient of friction was obtained.

RESULTS AND DISCUSSION

The test results will be discussed in the following order (1) Effect of resin-pigment ratio, (2) Effect of film thickness and (3) Effect of Wear.

A. EFFECT OF RESIN-PIGMENT RATIO

The solid film lubricant material, developed at Rock Island Arsenal and meeting the test requirements of Military Specification MIL-L-46010(MR), contains 27.3% epoxy-phenolic resin by weight based on the cured coating. When this material was originally formulated, the pigments, molybdenum disulphide, antimony trioxide and dibasic lead phosphite, were dispersed by means of a ball mill. The cured coating produced by above formulation had a Falex Wear life of 365 minutes at the standard 1000 lb. test load and a Salt Fog failure time of 24 hours. When this same solid film lubricant was produced by a paint manufacturer using a high shear rate type apparatus for dispersing the pigment, an increase in wear life and salt fog life occurred. The wear life was increased from the previous value of 365 minutes to a new value of 500 minutes, likewise, salt fog life was increased from 24 hours to 100 hours.

The difference in results can be attributed to the fact that by using a high shear rate type dispersing apparatus, the agglomerates of pigments are broken up

much more thoroughly than by simple ball milling. This, of course, results in much better pigment dispersion. This better pigment dispersion produced a more uniform solid film lubricant coating, thus increasing its wear life and corrosion preventing properties.

Since this more efficient pigment dispersion had a quite significant effect on wear and salt fog life at one resin concentration the effect of pigment dispersion at several levels of resin concentration was investigated. This study was undertaken with the hope that through better pigment dispersion additional resin could be added to the formulation thus increasing corrosion protection and still produce a solid film lubricant that would pass the 450 minute wear life requirement of Specification MIL-L-46010(MR).

Two series of solid lubricants were formulated, one in which the pigments were poorly dispersed and the other series had good pigment dispersion. In Table I the percent by weight of epoxy phenolic resin in the cured coating is given along with the corresponding pigment volume concentration. The pigment volume concentration (PVC) in the cured coating was calculated from the resin and pigment weight by the standard method of the paint industry using the specific gravity and bulking values of the pigment and resin. (8)

The relationship between PVC and the amount of resin in the coating is as follows, as the PVC number decreases the percent resin increases as shown in Table I. It is assumed that the corrosion protection of the coating is mostly dependent on the percent resin in the coating. On the other hand wear life is more dependent on PVC. The validity of these assumptions will be determined by wear and corrosion test data.

The results of the Falex Wear tests and 20% Salt Fog tests on these two series of formulations are given in Tables II and III. The results for each test are graphically illustrated in Figures 1 and 2. The pigments used in all the formulations were molybdenum disulphide, antimony trioxide and dibasic lead phosphite.

The critical pigment volume concentration (CPVC) was determined for both series of formulations from the salt spray data and is shown in the tables as the asterisk value.

The critical pigment volume concentration has been defined by Asbeck and Van Loo⁽⁷⁾ as a fundamental transition point in a pigment binder system in which the appearance and behavior of the paint changes considerably.

TABLE I

**PIGMENT VOLUME CONCENTRATIONS FOR SOLID
FILM LUBRICANT COATINGS**

<u>% Resin in Coating</u>	<u>Pigment Volume Concentration (PVC)</u>	
	<u>Poor Pigment Dispersion</u>	<u>Good Pigment Dispersion</u>
10.0	68.8	
20.0	50.8	
27.3		39.1
30.5	35.5	
31.5		34.2
35.0		
36.0	30.1	
36.6		29.4
37.6		28.5
38.6		27.2
39.6		26.8
40.0	26.6	
40.6		26.0
41.6		25.3
50.0	19.6	
55.9		15.9

TABLE II

**TEST RESULTS FOR SOLID FILM LUBRICANT SERIES
WITH POOR PIGMENT DISPERSION**

<u>Pigment Volume Concentration</u>	<u>Falex Wear Life (Minutes)</u>	<u>20% Salt Fog Life (Hours)</u>
68.8	215	6
50.8	280	16
35.5	390	32
30.1	410	90
26.6	335	120
*19.6	0	400

***Critical Pigment Volume Concentration (CPVC)**

TABLE III

**TEST RESULTS FOR SOLID FILM LUBRICANT SERIES
WITH GOOD PIGMENT DISPERSION**

<u>Pigment Volume Concentration (PVC)</u>	<u>Falex Wear Life (Minutes)</u>	<u>20% Salt Fog Life (Hours)</u>
39.1	500	100
34.2	550	120
29.4	695	300
*28.5	800	1200
27.2	715	-
26.8	620	-
26.0	73	-
25.3	40	-
15.9	0	-

***Critical Pigment Volume Concentration (CPVC)**

As shown by the data in Tables II and III and graphically in Figure 2 there is a sharp increase in salt fog life at the CPVC value. For both dispersions, the salt fog life at the CPVC is three to four times that at the next highest PVC evaluated. The CPVC value for the poor dispersion of 19.6 is lower than the CPVC value for the good dispersion of 28.5, this is in agreement with Asbeck and Van Loo⁽⁷⁾ who state that the CPVC for an agglomerate system is less than that for a dispersed system.

The test data shows that Falex Wear life increases with decreasing PVC (increasing resin) until a maximum value is reached after which the wear life decreases sharply. This sharp decrease is due to the fact that the PVC has become so low that there is not enough pigment present to support the 40,000 PSI test load at which the test is run. It should be noted that for the good dispersion series, the maximum wear life occurs at the CPVC. This can be expected as the CPVC is also defined as that point in a pigment vehicle system at which just sufficient resin is present to completely fill the voids left between the pigment particles in the film after the volatilization of the solvent. Therefore, if there is insufficient resin some of the voids between the pigments aren't filled so that the pigment is not tightly bound and, therefore, can wear away quite rapidly. On the other hand, if there is an excess of resin, sufficient pigment is not present so that the high test load can not be supported and the film fails rapidly.

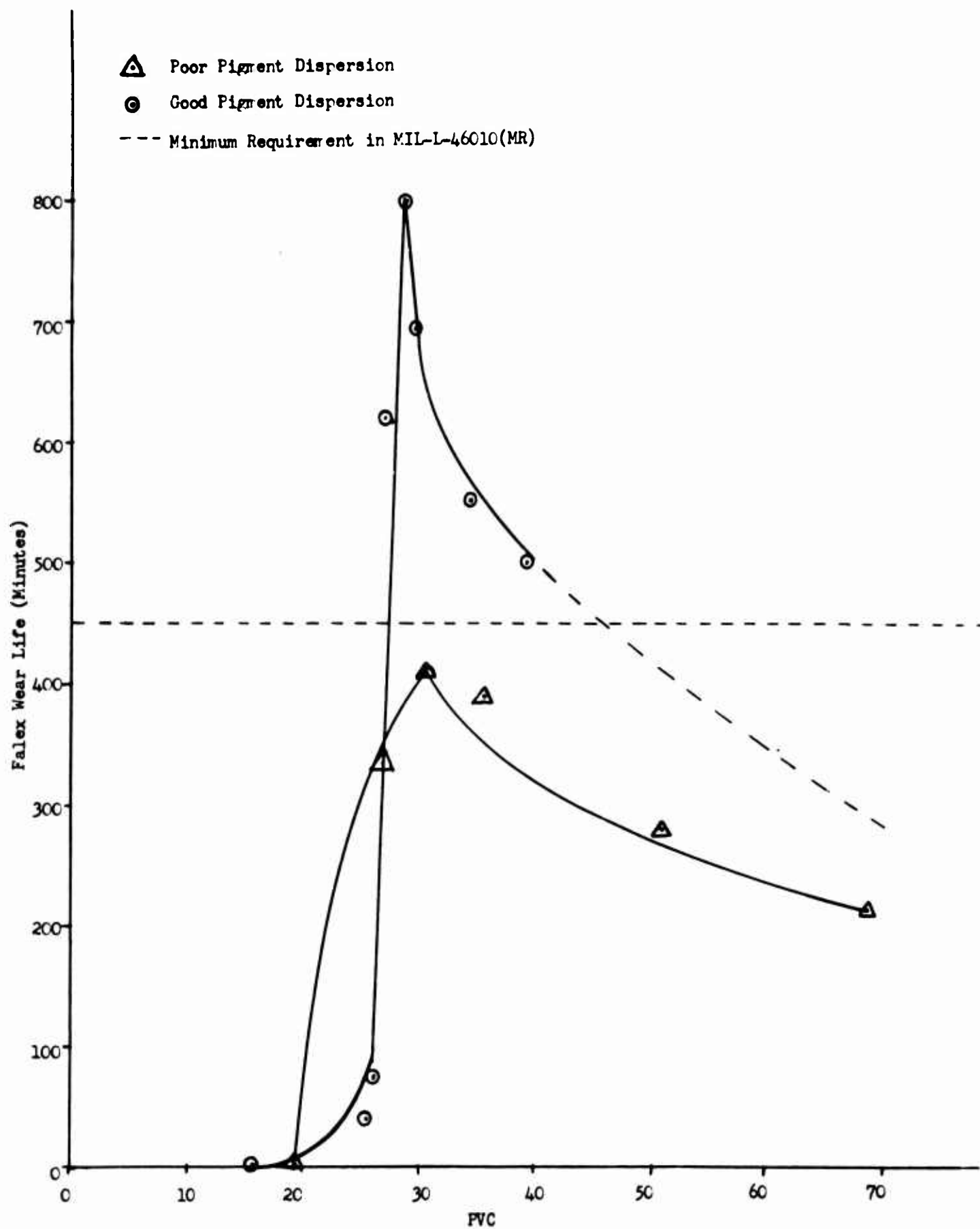


FIGURE 1

FALEX WEAR LIFE VS PVC

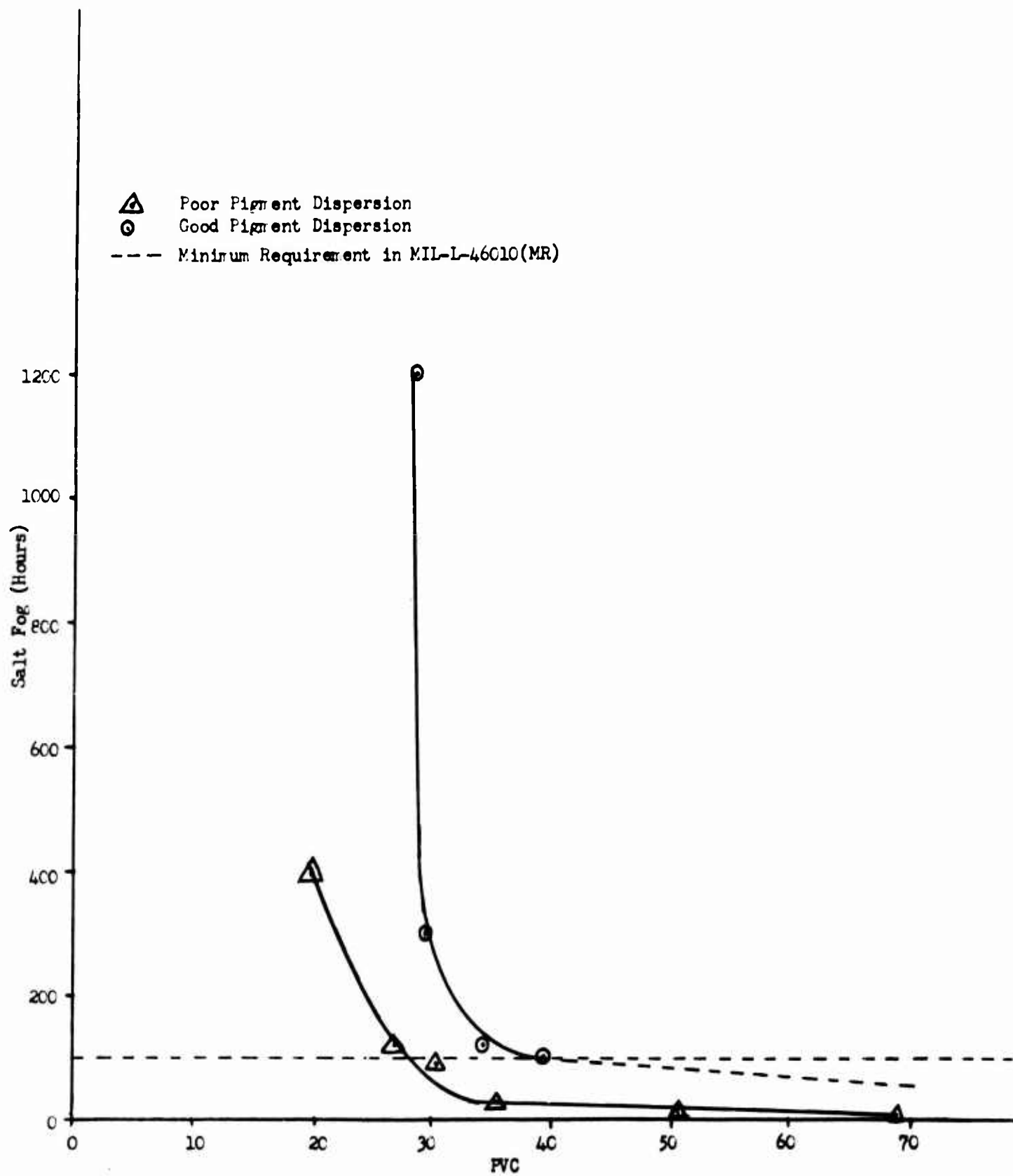


FIGURE 2

SALT FOG LIFE VS PVC

In the case of the poor dispersion series the CPVC value is so low that so much resin is necessary to just fill the voids between the pigments that there isn't enough pigment to support the test load. Therefore the coating fails as soon as the test load is reached. The maximum wear life is thus obtained at a PVC value that gives relatively poor corrosion protection.

The PVC value at which maximum wear life occurs is, therefore, dependent on the degree of dispersion. The better the dispersion the greater the amount of binder that can be present before the decrease in wear life becomes apparent. This accounts for the much longer corrosion protection at maximum wear life for the good dispersion series.

The dotted horizontal line in Figures 1 and 2 represent the test requirement for the test in Military Specification MIL-L-46010(MR). The minimum test values are 450 minutes of wear life and 100 hour salt fog life. As shown in Figure 1 the wear life requirement can not be met by a poorly dispersed formulation. Salt fog tests were not run on the good dispersion series formulations whose PVC value was 27.2 or lower, however, it can be expected that the salt fog life would increase greatly with decreasing PVC. This information would be of little practical value because for PVC values below 26.8 the wear life would be little or nothing thus there would no longer be a solid film lubricant per se.

From the data in Tables II and III and also Figures 1 and 2 it has been shown that by using the exact balance of pigment resin ratio and degree of dispersion a solid film lubricant can be produced that provides a large increase in corrosion protection without a sacrifice in wear life. If anything, the wear life has been increased. This, in part, solves the problem of increasing the corrosion protective properties of MIL-L-46010(MR) materials without sacrificing wear life.

It can also be stated that this solid film lubricant containing a PVC of 28.5, that was manufactured by means of good pigment dispersion techniques, is vastly superior to materials obtained commercially under Military Specification MIL-L-8937(ASG). The Falex Wear Test and 20% salt fog test results of the qualified MIL-L-8937(ASG) materials are given in Table IV. These tests were run on specimens that had been zinc phosphatized after grit blasting. The coating thickness was 0.0005 in. and coatings were cured at the time and temperature recommended by the manufacturer.

TABLE IV

TEST RESULTS FOR MIL-L-8937(ASG) SOLID FILM LUBRICANTS

<u>Solid Film Lubricant</u>	<u>Falex Wear Life (Minutes)</u>	<u>20% Salt Fog Life (Hours)</u>
1	80	<4
2	230	<4
3	136	4
4	168	<4
5	210	4
6	90	67
7	60	65
8	84	2
9	240	16

B. EFFECT OF FILM THICKNESS

Another factor that could affect the corrosion protection provided by solid film lubricant coatings is the film thickness of the coating. Ideally, for good corrosion protection the film thickness should be several mils thick such as is obtained when applying paint.

Unfortunately increased film thickness has an effect on the wear life of solid film lubricant coatings. Crump⁽⁶⁾ recommends an optimum initial thickness of 0.0004 in. and Palmer⁽⁹⁾ recommends a film thickness of 0.0002-0.0006 for optimum wear life. Both of them show that wear life decreases when film thickness greater than these recommended ones are used.

These recommendations were made for solid film lubricants containing either molybdenum disulphide or a combination of molybdenum disulphide and graphite as the lubricating pigment. Since the lubricating pigment system used in this investigation differed from those mentioned above, the effect of film thickness on the wear life of the Rock Island Arsenal system was investigated.

The effect of film thickness on wear life is shown in Table V for the 28.5 PVC formulation that was manufactured using good pigment dispersing techniques.

TABLE V
**EFFECT OF FILM THICKNESS ON WEAR LIFE OF A
 SOLID FILM LUBRICANT**

<u>Film Thickness (Inches)</u>	<u>Wear Life (Minutes)</u>
0.0005	800
0.0010	722
0.0013	191

This data agrees with Crump and Palmer in the sense that wear life does decrease with increasing film thickness. However, it also shows that a film thickness of 0.0010 inches, which is greater than that recommended by Crump and Palmer, can be used and still obtain a wear life that surpasses the wear life requirement of Specification MIL-L-46010(MR). This specification has the most stringent wear life requirement of any existing solid film lubricant specification.

The data also shows care must be taken in applying the solid film coating, as a thickness only slightly greater than the optimum thickness causes a sharp reduction in wear life.

Salt fog tests were run for two thicknesses of film for several formulations from the good pigment dispersion series. The results of these salt fog tests are shown in Table VI.

TABLE VI
**EFFECT OF FILM THICKNESS ON CORROSION
 PROTECTION PRODUCED BY SOLID FILM LUBRICANTS**

<u>Pigment Volume</u>	<u>Film Thickness (Inches)</u>	<u>Salt Fog Life (Hours)</u>
39.1	0.0005	100
39.1	0.0010	625
29.4	0.0005	300
29.4	0.0010	8200 ⁺
28.5	0.0005	1200
28.5	0.0010	7750 ⁺

The data in the above table points up the fact that doubling the coating thickness increases the corrosion preventing properties of the solid film lubricant coating by a factor of at least six at all PVC levels. It also shows that for any film thickness level corrosion protection increases with decreasing PVC.

By proper balancing of pigment-resin ratio, degree of dispersion and film thickness a solid film lubricant coating can be produced that has phenomenal corrosion protecting ability without sacrificing long wear life. Of course, this doesn't mean that the optimum coating has been obtained. It is quite possible that a more efficient combination of lubricative pigments could be developed that would allow the addition of more binder while still maintaining long wear life. This increased amount of resin would, of course, increase the corrosion protecting ability of the coating considerably.

In addition to the salt fog test, the effect of PVC and coating thickness on the corrosion preventing abilities of solid film lubricant coating was also evaluated by means of outdoor exposure tests. In these tests panels that had been given various pretreatments were coated with the 39.1 and 28.5 PVC good dispersion formulations at film thickness levels of 0.0005 and 0.0010 inches. The panels were subjected to outdoor exposure conditions at Rock Island Arsenal and the Fort Sherman test site in the Panama Canal Zone.

For this report, we will consider only the zinc phosphatized-SAE 1009 steel panels coated with the solid film lubricants. At the end of 8 months at the Rock Island Arsenal test site no comparison can be made as no corrosion has appeared on the panels. The test at Panama with its more severe conditions has given some results after only two weeks exposure. The test results show that the 28.5 PVC coated panels are not corroded at either film thickness. The 39.1 PVC coated panels show considerable corrosion for both film thickness. The corrosion for the 0.0010 in. coating is not as extensive as that for the 0.0005 in. coating. This data agrees with the salt fog data.

C. EFFECT OF COATING WEAR

Previous work by Calhoun et al⁽⁵⁾ has shown that rusting of surfaces coated with solid film lubricants has little affect on the wear life of a good corrosion inhibiting solid film lubricant. For example, the wear life of the 39.1 PVC solid film lubricant only decreased about 15% after 120 days exposure in the 20% salt fog

cabinet prior to testing. This exposure resulted in rust on 50% of the specimen surface.

Since little work has been done on the effect of wear on the corrosion inhibiting ability of a solid lubricant coating, several coatings were evaluated in a combination friction-corrosion test procedure previously described. The results of the friction-corrosion test are given in Table VII. Coefficient of friction was determined under a load of 15 psi at a speed of 3/4 inch per minute.

From the data in the tables it is evident that the time required to reach 100% increase in the coefficient of friction is related to the original corrosion preventive properties of the solid lubricant coating. Lubricant #2, whose coefficient of friction doubled after the shortest salt fog exposure time, has very poor initial corrosion preventing properties as shown in Table IV. The 28.5 PVC lubricant on the other hand which required the longest salt fog exposure time for doubling the coefficient of friction has excellent initial corrosion preventing properties. The other two lubricants, #6 and the 39.1 PVC, are intermediate both as to exposure time and original corrosion protective properties.

The effect of pigment volume concentration is quite evident in this test. After 720 hours salt fog exposure the coefficient of friction increased very little for the 28.5 PVC formulation while it doubled for the 39.1 PVC formulation. The 28.5 PVC formulation also did a much better job of protecting against corrosion than did the higher PVC formulation. This data also shows that the 28.5 PVC formulation is superior to the best corrosion preventing MIL-L-8937(ASG) material.

Although a certain degree of corrosion protection can be obtained for lubricated parts in actual use by using a good solid film lubricant a more efficient one is something that should be developed.

CONCLUSIONS

The following conclusions can be made from this study: (1) both the resin-pigment ratio and the degree of pigment dispersion effect the corrosion preventive properties of solid film lubricant coatings. (2) By a proper balance of resin and pigment and degree of pigment dispersion, a solid film lubricant was developed that has corrosion preventing properties far in excess of those obtained from the present MIL-L-8937(ASG) or MIL-L-46010(MR) materials. This improved corrosion inhibiting property was obtained at no sacrifice to wear life. (3) Increasing

TABLE VII
FRICTION-CORROSION TEST

Hours in Salt Fog	Lubricant 2			Lubricant 6			39.1 PVC Lubricant			28.5 PVC Lubricant		
	% Rust	μ^*	% Change in μ	% Rust	μ^*	% Change in μ	% Rust	μ^*	% Change in μ	% Rust	μ^*	% Change in μ
0	-	0.14	-	-	0.13	-	-	0.15	-	-	0.19	-
72	100	0.16	+14.3	40	0.18	+38.5	10	0.14	-6.7	5	0.15	-21.0
144	100	0.19	+35.7	60	0.17	+30.8	15	0.14	-6.7	5	0.18	-5.3
216	100	0.35	+150.0	80	0.19	+46.2	40	0.16	+6.7	10	0.17	-10.5
288	100	0.34	+142.8	90	0.18	+38.5	40	0.19	+26.7	10	0.16	-15.8
360				95	0.14	+7.1	50	0.21	+40.0	15	0.16	-15.8
432				95	0.18	+38.5	70	0.21	+40.0	15	0.18	-5.3
504				100	0.18	+38.5	90	0.24	-60.0	15	0.18	-5.3
576				100	0.21	+61.5	100	0.26	+73.3	20	0.11	-42.1
648				100	0.24	+84.6	100	0.24	+60.0	25	0.20	+5.3
720				100	0.26	+100.0	100	0.32	+100.0	30	0.20	+5.3
792										40	0.23	+21.0
1080										60	0.23	+21.0
1152										60	0.27	+42.1
1224										80	0.33	+73.0
1440										80	0.38	+100.0

* μ = coefficient of friction

the coating thickness from 0.0005 to 0.0010 inches causes a sixfold increase in the corrosion inhibiting abilities of the solid film lubricant coating. (4) The effect that wearing away of the lubricant coating during use has on its ability to prevent corrosion depends on the original corrosion inhibiting properties of the solid film lubricant coating. The better the original protection the less the effect of coating wear or corrosion resistance.

RECOMMENDATIONS

1. It is recommended that the 28.5 PVC solid film lubricant replace the present MIL-L-46010(MR) material.
2. It is further recommended that the thickness of the solid film lubricant coating be limited to a maximum value of 0.0010 inches.
3. Finally, it is recommended that work be done to improve the corrosion inhibiting properties of solid film lubricants while they are in use.

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13. ABSTRACT This investigation included the effect of (1) resin-pigment ratio, (2) coating thickness, and (3) wearing away of the film on the corrosion protection provided by solid lubricant coatings. These factors were investigated as possible approaches to improvement of the corrosion inhibition provided by MIL-L-46010(MR) type solid film lubricants. This improved corrosion inhibition was to be obtained at no sacrifice in wear life. Two series of solid film lubricants of various resin-pigment ratios were made using good and poor pigment dispersion techniques. These series of solid film lubricants were evaluated at several levels of coating thickness. Corrosion protection was evaluated by means of 20% Salt Fog cabinet while wear life was determined by the Falex Wear Test method. A combination friction-salt fog test was used in determining the effect of wearing away of the film on corrosion resistance. It was found that a solid film lubricant can be manufactured, using good pigment dispersion techniques, which will provide 1000 hours of salt fog protection. This lubricant showed no loss in wear life. It was also found that, by using a 0.0010 in. coating made up of two layers, salt fog life can be increased by at least 6 fold without sacrificing wear life. The investigation also pointed up the fact that wearing away of the solid lubricant coating decreases the effectiveness of the coating as a corrosion inhibiting barrier. (U) (Author)		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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